COMPONENTS:

- (1) Chromium: Cr: [7440-47-3]
- (2) Mercury; Hg: [7439-97-6]

EVALUATOR:

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July, 1985

CRITICAL EVALUATION:

The solubility of chromium in mercury near room temperature is very low. Irvin and Russell (1) reported that it is below their analytically detectable limit of 2 x 10^{-4} at % at 293 K, while DeWet and Haul (2) reported that it is below 1.5 x 10^{-6} at % at 303 K. Jangg and Palman (3) extrapolated their high temperature measurements and estimated a solubility of 7.7 x 10^{-7} at % at 293 K. The solubility of 1.2 x 10^{-10} at % at 291 K, reported by Tammann and Hinnüber (4) from EMF measurements, is too low, while the solubility of 7 x 10^{-3} at % at room temperature, reported by Strachan and Harris (5), is much too high. Kozin (6) predicted a solubility of 5.2 x 10^{-4} at % at 298 K.

High temperature solubility measurements have been reported by several authors. Jangg and Palman (3) determined solubilities of 2.6 x 10^{-4} and 3.1 x 10^{-4} at % at 773 and 823 K, respectively, and they estimated solubilities at lower temperatures by extrapolating the two high temperature values; the solubilities obtained from this extrapolation appear to be dubious. Weeks (7), without presenting experimental details, reported a solubility of 2.6 x 10^{-3} at % at 873 K. Weeks and Fink (8-10) extended the solubility measurements over the temperature range of 778 to 923 K and these authors reported their results in graphical form; the only numerical value reported was 1.1 x 10^{-2} at % at 923 K (10). Parkman (11) reported a solubility of 2.1 x 10^{-4} at % at 866 K; the latter result is in better agreement with that of (3) than with (8).

As seen from the high temperature measurements, the solubility of chromium in mercury is very low over a wide temperature range. There is some disagreement in the high temperature measurements reported by refs. (3), (7), (8-10), and (11), and a plot of the logarithm of solubility against reciprocal temperature shows different slopes for these sets of data.

The liquid amalgam is in equilibrium with pure chromium, and no intermetallic compounds are formed with mercury (2, 12).

Solubilities of Cr-containing alloys in mercury were reported in (11, 13, 14).

The tentative value for the solubility of chromium in mercury at 773 K is 2×10^{-4} at %; this value is the mean from refs. (3) and (8).

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Chromium

COMPONENTS: (1) Chromium; Cr; [7440-47-3] de Wet, J.F.; Haul, R.A.W. (2) Mercury; Hg; [7439-97-6] Z. Anorg. Chem. 1954, 277, 96-112. VARIABLES: One temperature: 303 K PREPARED BY: C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of chromium in mercury at 303.2 K was reported to be below 4 x 10^{-7} mass %. The corresponding atomic % solubility limit calculated by the compilers is 1.5 x 10^{-6} at %.

Pure chromium was found to be in equilibrium with mercury.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Heterogeneous amalgam was obtained by electrolysis of Cr(III)-sulfate at the mercury cathode. The amalgam was washed, dried and sealed off under vacuum in glass ampules. Later, a portion of the amalgam was centrifuged and 10-12 g of the homogeneous amalgam was taken for analysis. The mercury was carefully distilled off and the residue was dissolved in HC1. The resulting solution was analyzed spectrochemically for chromium.

SOURCE AND PURITY OF MATERIALS:

 $Cr_2(SO_4)_3$ ·18 H_2O was BDH Lab. Reag.

Purified Hg was distilled under vacuum and was found to be spectrochemically free of chromium.

All other chemicals and apparatus were carefully cleaned.

ESTIMATED ERROR:

Detection limit for Cr was 4×10^{-7} mass %. Temp: nothing specified.

EXPERIMENTAL VALUES:

The solubility of chromium in mercury:

<u>t/°C</u>	Soly/mass %	Soly/at % ^a
550	8.0×10^{-5}	3.1×10^{-4}
500	6.8×10^{-5}	2.6×10^{-4}
400 ^b	3.3×10^{-5}	1.3×10^{-4}
300 ^b	2×10^{-5}	7.7×10^{-5}
200 ^b	7.2×10^{-6}	2.8×10^{-5}
100 ^b	1.5×10^{-6}	5.8×10^{-6}
20 ^b	2×10^{-7}	7.7×10^{-7}

 $^{^{}a}$ by compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The heterogeneous amalgam was introduced into specially constructed apparatus made of refractory chromium steel. Such steel apparatus could be used because the solubility of iron in mercury is very low and the Cr(III)-oxide film inhibits the wetting of the steel by mercury. After 12 hr of equilibration at the temperature of the experiment, the amalgam was filtered through a sintered iron frit in an atmosphere of purified nitrogen. The chromium content in the filtered, saturated amalgam was determined by an unspecified method.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: accuracy \pm 5%. Temp: precision \pm 2 K.

^bOnly the 500 and 550°C solubilities were experimentally determined; the values at lower temperatures were estimated by extrapolation of the two experimental values.

COMPONENTS: (1) Chromium; Cr; [7440-47-3] Parkman, M.F. (2) Mercury; Hg; [7439-97-6] Ext. Abst., Electrothermics and Metallurgy Div., The Electrochemical Soc., Vol. 2, No. 2, 1964, pp. 16-21. VARIABLES: PREPARED BY: One temperature: 866 K C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The mass % solubility of chromium in mercury was reported graphically; a value of 5.5 x 10^{-5} mass % at 866 K was read off the curve by the compilers. The corresponding atomic % solubility calculated by the compilers is 2.1 x 10^{-4} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Specimen of Cr was placed in contact with Hg in a glass capsule. The capsule was sealed under vacuum after at least 16 hr outgassing of the Hg. The temperature of the capsule was raised to the desired level and held there for 16 hr. A sample of the solution was then collected and cooled. Hg was separated from the sample by molecular distillation. The residue was taken into acid solution, dried, and analyzed by emission spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Pure chromium from AGN.

Mercury purity not specified, but probably triple-distilled.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision + 3 K.

COMPONENTS: (1) Chromium; Cr; [7440-47-3] Weeks, J.R. (2) Mercury; Hg; [7439-97-6] Corrosion 1967, 23, 98-106. VARIABLES: Temperature: 505-650°C PREPARED BY: C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The mass % solubility of chromium in mercury was presented graphically as a function of temperature. The data points were read from the curve and the solubility converted to atomic % by the compilers.

<u>t/°C</u>	Soly/at %
650 ^a	1.1 x 10 ⁻²
605	4.6×10^{-3}
575	9.6 x 10 ⁻⁴
555	5.0×10^{-4}
550	4.2×10^{-4}
530	3.6×10^{-4}
510	2.5×10^{-4}
505	1.2×10^{-4}

^aThis value also reported in (1); the other data also reported in (2).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Mercury and chromium were sealed in the upper chamber of an evacuated quartz tube. The two chambers were separated by a coarse quartz filter. The filled tubes were equilibrated for 72 hr at each temperature, then centrifuged at temperature to force the liquid through the filter. The mercury was distilled from the known quantity of amalgam, and the residue was dissolved in HF-HNO3 or aqua regia and analyzed spectrographically.

SOURCE AND PURITY OF MATERIALS:

Mercury was triple-distilled. Chromium purity not specified.

ESTIMATED ERROR:

Soly: nothing specified. Temp: precision \pm 2 K.

- Weeks, J.R.; Fink, S. U.S. At. Ener. Comm. Rep., BNL-900, 1964, pp. 136-9.
- Same authors
 U.S. At. Ener. Comm. Rep., BNL-782, 1962, pp. 73-5.

COMPONENTS:

- (1) Molybdenum; Mo; [7439-98-7]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski: Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

Published experimental values show low solubility of molybdenum in mercury. Tammann and Hinnüber (1) could not detect any dissolution of molybdenum, and Iryin and Russell (2) concluded that the solubility at 293 K should be lower than 4×10^{-5} at %. Strachan and Harris (3) could not detect any molybdenum in mercury by their analytical procedure which had a detection limit of 2×10^{-3} at %. Kozin first predicted a solubility of 7.5 x 10^{-20} at % at 298 K (4), but this was later revised to an estimate of 2.5 x 10^{-13} at % (5). Based on regular solution theory, Brewer and Lamoreaux (6) derived the equation.

 $\ln N = 3 - 20000/(T/K)$

where N is the solubility of molybdenum in at %; the solubility calculated at 298 K from this equation is 10^{-28} at %.

At high temperatures, Bowersox and Leary (7,8) reported that the solubility is lower than 1.5 x 10^{-5} at % at 623 K, while Messing and Dean (9) found that the solubility of molybdenum in saturated uranium amalgam is also below the detection limit of 1.1 x 10^{-4} at % at 629 K.

No corrosion of Mo by Hg was observed after their contact at 719 K for more than 30 days (10). This means that the solubility of Mo in Hg should be very low, probably of similar order of magnitude as for Nb.

Férrée (11) reported the intermetallic compounds, MoHgg, Mo2Hgg and MoHgg, but these results are questionable and further experimental work is needed on this system.

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COMPONENTS:	ORIGINAL MEASUREMENTS:	
(1) Molybdenum; Mo; [7439-98-7] (2) Mercury; Hg; [7439-97-6]	Bowersox, D.F.; Leary, J.A. U.S. At. Ener. Comm. Rep., LAMS-2518, 1961.	
VARIABLES:	PREPARED BY:	
One temperature: 350°C	C. Guminski; Z. Galus	

EXPERIMENTAL VALUES:

The solubility of Mo in Hg at 350°C is lower than 0.001 g of Mo in 1 dm 3 of Hg. The corresponding solubility limit calculated by the compilers is 1.5 x 10 $^{-5}$ at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined by immersing a weighed coupon of molybdenum into a definite amount of boiling mercury and periodically measuring the coupon weight. Thus, the weight loss corresponds to the part of the molybdenum that dissolved.

SOURCE AND PURITY OF MATERIALS:

Triple-distilled mercury was used. Molybdenum purity not specified.

ESTIMATED ERROR:

Soly: detection limit was 1 mg of Mo.

Temp: nothing specified.

COMPONENTS:

- (1) Tungsten; W; [7440-33-7]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION:

The solubility of tungsten in mercury is very low, and no accurate measurements have been reported. Irvin and Russell (1) have shown that the solubility at 293 K is lower than 1×10^{-5} at % and Strachan and Harris (2) could not detect tungsten in mercury at their detection limit of 10^{-3} at %. Tammann and Hinnüber (3) also could not detect the dissolution of tungsten in mercury. Raub and Plate (4) observed that there was no interaction between the two metals at 1273 K. Similarly, Nejedlik and Vargo (5) found that tungsten was inert to mercury after contact for more than 30 days at 719 K, thus indicating that the solubility of tungsten is very low. It is probable that the solubility of tungsten is of the same magnitude, or lower, as that of tantalum.

The low solubility of tungsten also is suggested by the semiempirical estimates of Kozin who reported values of 4.8 x 10^{-33} (6) and 6.8 x 10^{-20} at % (7) at 298 K.

References

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 Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii, Nauka, Alma-Ata, <u> 1964.</u>